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COMPARATIVE STUDY Of THE ESSENTIAL OILS OF *Heracleum sphondylium* ssp. *ternatum* OBTAINED BY MICRO- AND HYDRO-DISTILLATION METHODS*

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The volatile compounds obtained by hydro- and micro-distillation methods from crushed fruits of Heracleum sphondylium *ssp.* ternatum *(Velen.) Brummitt were analyzed by GC/MS. Both distillation methods and analysis results were compared.*

Key words: *Umbelliferae*, *Heracleum sphondylium* ssp. *ternatum*, essential oil, micro-distillation, GC/MS.

The genus *Heracleum* (*Umbelliferae*) comprises more than 70 species [1]. This genus is represented in Turkey by 14 species, 7 of them being endemic [2]. *H. sphondylium* is known as «Tavsancilotu» and is used against dysentery and diarrhea [3]. The volatile fraction of a petroleum extract of the seed of *H. sphondylium* was investigated. *n*-Octyl acetate and *n*-octyl caproate were found as major constituents [4].

Although the essential oil composition of various members of this genus have been reported [5-9], *H. sphondylium* L. ssp. *ternatum* (Velen.) Brummitt (Syn: *H. ternatum* Velen.) has not been investigated previously.

Some biological activity studies of *H. sphondylium* L. and *H. sphondylium* L. ssp. *ternatum* (Velen.) Brummitt have also been reported [9, 10].

The crushed fruits of *H. sphondylium* ssp. *ternatum* (Velen.) Brummitt were hydro- and micro-distilled to yield volatile compounds which were investigated by GC/MS. The identified components with their relative percentages are given in Table 1.

In the hydro-distilled oil of *H. sphondylium* ssp. *ternatum* 38 compounds representing 97.2% of the total oil were characterized, with octanol 39.2%, octyl butyrate 27.4%, and octyl acetate 10.6% as the major components. In the micro-distilled sample 32 compounds representing 97.2% of the total oil were characterized. Octyl butyrate 42.9%, octyl acetate 30.9%, and octanol 9.0% were found as the main components.

It is interesting to note that in the hydro-distilled oil the octyl acetate and octyl butyrate amounts are lower than in the micro-distilled sample, due to hydrolysis of octyl esters in the hydro-distillation process. Therefore, the octanol amount is much higher in the hydro-distilled oil, whereas analysis of the micro-distillation sample suggests that no hydrolysis occurred and that micro-distillation is a milder method.

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TABLE 1. Comparative Results of the Essential Oils of *Heracleum sphondylium ssp. ternatum*

RRI	Compound	A(%)	B(%)
1048	2-Methyl-3-buten-2-ol	0.1	-
1241	Butyl-2-methylbutyrate	0.1	0.1
1246	(*Z*)-*β*-Ocimene	0.2	0.2
1259	Butyl 3-methyl butyrate (*Butyl isovalerate*)	0.3	-
1282	Hexyl acetate	0.1	Tr
1285	Isoamyl isovalerate	0.1	0.1
1296	Octanal	1.6	0.8
1299	2-Methylbutyl isovalerate	0.3	0.2
1327	3-Methyl-2-butenol	0.1	-
1353	Hexyl isobutyrate	0.1	0.1
1360	Hexanol	0.5	Tr
1400	Nonanal	0.2	0.1
1424	Hexylbutyrate	0.4	0.3
1438	Hexyl-2-methyl butyrate	0.5	0.6
1457	Hexyl-3-methyl butyrate (*Hexylisovalerate*)	1.0	1.0
1483	**Octyl acetate**	**10.6**	**30.9**
1506	Decanal	0.2	0.2
1516	(*Z*)-4-Octenyl acetate	0.6	2.3
1546	(*Z*)-4-Decenal	0.1	-
1547	Octyl isobutyrate	0.3	0.2
1562	**Octanol**	**39.2**	**9.00**
1589	(*Z*)-3-Octen-1-ol	3.0	0.6
1623	**Octyl butyrate**	**27.4**	**42.9**
1634	Octyl-2-methyl butyrate	0.8	0.4
1654	Octyl-3-methyl butyrate (*Octylisovalerate*)	0.6	0.4
1660	(*Z*)-4-Octenylbutyrate	1.6	2.1
1672	(*Z*)-4-Octenyl-2-methyl butyrate	Tr	0.1
1687	Decyl acetate	0.2	0.5
1727	Nonyl butyrate	-	0.1
1766	Decanol	0.5	0.1
1783	*β*-Sesquiphellandrene	0.1	0.1
1829	Octyl hexanoate	1.6	1.0
1830	Decyl butyrate	0.3	0.3
1856	(*Z*)-4-Octenylhexanoate	0.4	0.3
2020	Octyl octanoate	0.2	-
2239	Carvacrol	0.1	0.1
2296	Myristicine	3.2	2.1
2594	9-Hexacosene	0.1	-
2713	Tetradecanoic acid	0.5	-
	Total	**97.2**	**97.2**

A: Hydro-distillation.

B: Micro-distillation.

RRI: Relative retention indices on a polar column.

Tr: Trace (< 0.1%).

EXPERIMENTAL

Plant Material. Plant material was collected from Denizli, Elmayani village, on 12 July 1999. Voucher specimens are kept at the Herbarium of the Faculty of Pharmacy (ESSE 12874), Anadolu University, Eskisehir, Turkey.

Distillation Method. The essential oils were obtained by hydro-distillation and micro-distillation from crushed fruits of *H. sphondylium* L. ssp. *ternatum* (Velen.) Brummitt. For the micro-distillation we used an Eppendorf MicroDistiller®. The oils were analyzed by GC/MS.

Hydro-distillation. Air dried and crushed fruits of *H. sphondylium* ssp. *ternatum* was hydrodistilled for 3 h using a Clevenger-type apparatus. The yield was calculated on a dry weight basis, 3.7%

Micro-distillation: Dried and crushed fruits (~500 mg) were placed in a sample vial together with 10 ml of water. NaCl (2.5 g) and water (0.5 ml) were placed in the collecting vial. *n*-Hexane (300 μL) was added to the collecting vial to trap volatile components. Sample vials were heated to 108°C at a rate of 20°C/min, kept at 108°C for 90 min, heated to 112°C at a rate of 20°C/min, and kept at this temperature for 30 min. Finally the samples were subjected to a post-run for 6 min under the same conditions. Collecting vials were cooled to -1°C during distillation. After the distillation was completed, the organic layer in the collection vial was injected into the GC/MS instrument.

Analysis of Essential Oils. The essential oils were analyzed using a Hewlett-Packard G1800A GCD system with Innowax FSC column (60 m × 0.25 mm ∅, with 0.25 μm film thickness). Helium (0.8 mL/min) was used as carrier gas. The GC oven temperature was kept at 60°C for 10 min and programmed to 220°C at a rate of 4°C/min and then kept constant at 220°C for 10 min and programmed to 240°C at a rate of 1°C/min. The Mass range was recorded from *m/z* 35 to 425. The split ratio was adjusted at 50:1 for the hydro-distilled sample. Injection of the micro-distilled sample was applied splitless. The injection port temperature was at 250°C. MS were recorded at 70 eV. Relative percentage amounts of the separated compounds were calculated automatically from peak areas of the total ion chromatogram. Alkanes were used as reference points in the calculation of relative retention indices (RRI). Library search was carried out using the "Wiley GC/MS Library" and the "TBAM Library of Essential Oil Constituents."

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